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MICROWAVE-ASSISTED PRODUCING CLOSED-CELL ALUMINUM FROTH

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ABSTRACT: This work constitutes a contribution of authors to making microwave-assisted closed-cell aluminum froth. The required aluminum powder was obtained as a result of the own method applying by nitrogen jet atomization of the molten metal through rapid microwave heating, the solidified aluminum grains being very fine (under 10 μm). Reducing the aluminum powder granulation influenced the foam denseness decrease in the range of 0.45-0.82 $g \cdot cm^{-3}$ and the increase of its porousness up to 80.8 %. Depending on the size of the cell structure, the compression strength varied between 3.8-7.3 MPa. The main application areas of these froths are energy and respectively, sound absorption, electromagnetic shielding, and vibration damping.

KEYWORDS: aluminum froth, closed-cell, microwave, high-strength, high durability.

1. INTRODUCTION

In general, metal foams have remarkable abilities in terms of the correlation between physical and mechanical properties such as: light weight, strength, compactness, durability, resistance to high thermal loads, high thermal energy absorption capacity, etc. These special features are favourable for the use of metal foams in several industrial fields, especially in the aerospace, automotive, and naval industries as well as foam core material for sandwich panels in construction [1, 2]. Applications of metal foams under low temperature conditions have proven to be appropriate in the case of aluminum foams [1, 3], which, incidentally, is the material type focused on in the current work.

According to the literature, aluminum foams fall into two categories in terms of cell morphology: open-cell and closed-cell foams, respectively. The second foam category was investigated in this study. Closed-cell foams have a much lower effective surface area compared to open-cell foams, being particularly suitable for energy absorption [4], foam-core sandwich panels, shock absorbers, acoustic insulators, etc.

Metal foams can be made by several techniques depending on the required features of the foam. The liquid metallurgy procedure allows the cellular structure to be obtained directly from the molten metal. This method is much simpler compared to other methods and at the same time, it is very cost-effective [5]. Melts can be directly foamed by blowing a gas into the molten metal, or by adding an expanding agent, which thermally decomposes, releasing gases. The addition of refractory powders has the role of stabilizing the melt by increasing its viscosity.

Gas injection patented by Alcan International Ltd., Canada, involves melting metallic aluminum under the conditions of adding silicon carbide, alumina, titanium diboride, zirconium, and magnesium oxide powders with sizes between 5-20 μm. The gas (nitrogen, argon, or air) injected directly into the melt is introduced from the bottom up. In this way, very fine, homogeneously distributed gas bubbles are produced. The added powders represent between 10-20 % [6, 7].

Closed-cell metal foams have structures characterized exclusively by isolated pores. The compressive strength of these foams is higher compared to open-cell foams due to this structural peculiarity. In general, closed-cell foams have higher densities, explained by the higher amount of solid that composes the material matrix. Also, this type of metal foams has superior dimensional stability, higher strength, and lower moisture absorption capacity.

The method of producing metal froth in which expanding agents are introduced into the melt is also a procedure of froth making by the liquid metallurgy route, having the name of Alporas process [8]. The melt viscosity is increased through the addition of 1-2 % calcium as a thickening agent and 1-2 % hydrides such as TiH_2 or CaH_2 in the form of fine powders as an expanding agent. Alporas froth quality depends on the quality of powder mixing, melt viscosity, temperature, and cooling rate. The aluminum foam made by the Alporas method has relative densities in the range of 0.2-0.9.

The Gasar procedure is based on the following metal materials: copper, aluminum, nickel, magnesium, iron, and chromium [1, 9]. The use of a eutectic reaction in hydrogen-supersaturated melts generates porous structures. By cooling the melt, gaseous and solid phases are simultaneously formed. Solidification leads to increasing the hydrogen content at the solidification limit, causing the formation of gas bubbles. The process is controlled by the cooling rate of the melt, hydrogen pressure, and casting temperature of the melt. Despite the advantages offered by the possibility of applying the method to a wide variety of metals and obtaining many pore morphologies, the use of the Gasar procedure requires complex equipment, being considered quite unprofitable and also unsuitable for intensive manufacturing processes.

A recent technique for producing cellular metals is the powder metallurgy method designed and patented by the German Fraunhofer Institute [1]. Expanding agents are mixed into the metals, being introduced in the solid state. Titanium hydride (TiH_2), as a frequently used agent, thermally decomposes after 465 °C, representing a temperature much lower than the melting point of aluminum (660 °C). Thus, froth generation by dispersion of the agent in solid aluminum occurs using typical powder metallurgy processes. By increasing the temperature, gases are released into the partially or completely molten metal, leading to the development of bubbles. Subsequent cooling stabilizes the formed foam. This process is initiated by combining expanding agent particles with aluminum alloy powder. The powder is cold compacted and then extruded into a bar or plate. The extruded metal is cut into small pieces, placed in a closed die and heated to a temperature slightly above the solidus point of the alloy. TiH_2 decomposes forming voids at a high internal pressure. The aluminum mass increases in volume forming a foam that fills the interior space of the die, then stabilizes upon cooling. The relative density of the foam is very low (0.08). The diameter of the formed closed-cells is within the range of 1-5 mm. For now, the new method is expensive and not yet usable, despite the high quality of the foamed products.

A closed-cell aluminum froth manufacturing process was performed under original conditions by a team including authors of the current paper [10]. The experiment was carried out using recycled aluminum scrap from the commercial sector melted under the microwave action, the melt being atomized below 63 μm in contact with inert nitrogen jets into a metal enclosure with water-cooled walls. The fine aluminum powder was associated with the expanding agent (dolomite) as a substitute for the commonly used TiH_2. The making process varied between 705-735 °C, its duration being between 9.17-10.92 min corresponding to warming rate of above 65 $°C \cdot min^{-1}$. The features of the closed-cell aluminum froth were as follows: denseness between 1.16-1.19 $g \cdot cm^{-3}$, porousness between 55.9-57.0 %, compression strength in the range of 6.83-7.01 MPa, and heat conductivity within the limits of 5.71-5.84 $W \cdot m^{-1} \cdot K^{-1}$.

Another experiment of the Romanian authors' team carried out in 2023 [11] adopted $CaCO_3$ as an expanding agent, while the fine aluminum powder was also obtained by atomizing aluminum waste (below 32 μm) melted under the microwave action. The mixture created for the expanding process was warmed in a microwave-adapted oven at 750 °C. The main features of the aluminum froth were: denseness between 0.88-1.10 $g \cdot cm^{-3}$, heat conductivity in the range of 3.30-5.65 $W \cdot m^{-1} \cdot K^{-1}$, and compression resistance within the limits of 3.9-7.4 MPa.

2. MATERIALS AND METHODS

2.1 Materials

The aluminum froth manufacturing process was focused on using low-cost materials. Thus, calcium carbonate ($CaCO_3$) was chosen as an efficient and, at the same time, sufficiently cheap expanding agent, purchased from the market in the form of fine powder with grain size under 10 μm.

The aluminum as the basic raw material was manufactured by the own method [10, 11] using residual aluminum recovered from the commercial sector in the form of post-consumer metal cans. The waste was melted under the influence of microwave irradiation into a silicon carbide-crucible. Heated to temperatures around 750 °C, above the melting point of metal aluminum, the melt was freely flowed out from the crucible through a nozzle and brought into direct contact with several concentrated nitrogen jets in order to finely atomize it. The reception of small aluminum droplets was carried out in a metal chamber with walls intensely cooled with cold water to avoid agglomeration of several liquid droplets with solidified walls. The aluminum powder thus prepared had a remarkable fineness, the particle size being under 10 μm.

2.2 Methods

In terms of chemistry, the thermal decomposition reaction of $CaCO_3$ occurs in the temperature range of 800-900 °C [12], releasing CaO (solid) and CO_2 (gas), according to reaction (1).

$$CaCO_{3\,(solid)} \rightarrow CaO_{(solid)} + CO_{2\,(gas)} \qquad (1)$$

In the presence of molten aluminum above 700 °C, chemical reactions occur in the $CaCO_3$-liquid aluminum interface area, according to (2) and (3).

$$2Al_{(liquid)} + 3CO_{2\,(gas)} \rightarrow Al_2O_{3\,(solid)} + 3CO_{(gas)} \qquad (2)$$

$$8Al_{(liquid)} + 3CO_{2\,(gas)} \rightarrow 2Al_2O_{3\,(gas)} + Al_4C_{3\,(gas)} \qquad (3)$$

Reactions (2) and (3) can occur at temperatures even under the melting point of aluminum. In melts, the froth is formed due to CO_2 released from $CaCO_3$ only if the partial pressure of CO_2 inside the cell is maintained below its equilibrium value by reaction (2). The minimum required CO_2 (gas) to participate in the foaming the melt becomes possible under conditions in which about 30 % of the available $CaCO_3$ releases CO_2 [13].

The experiment involved dosing the two solid components of the mixture. Thus, $CaCO_3$ was distributed in the four experimental versions in different weight proportions: 3, 6, 9, and 12 %. Adopting the total amount of solids at 100 g, the dosages of aluminum powder had the following contents: 97, 94, 91, and 88 g. The mixture formed by aluminum powder and $CaCO_3$ wetted with 10 % water was pressed and introduced into a ceramic crucible made of SiC (80 %) and Si_3N_4 (20 %) originating in China. After protecting the outer surface of the crucible with ceramic fibre mattresses, it was placed in an 800 W-microwave oven of the type usually used in households for food preparation, but constructively and operationally adapted for operation at high temperatures (Figure 1). The temperature control of the heated material was performed with a radiation pyrometer mounted above the oven at about 400 mm, which can visualize the temperature through a 30 mm-hole drilled in the upper wall of the oven housing as well as in the ceramic lid of the crucible.

Figure 1. Overall image of the 800 W-microwave equipment

2.3 Investigation methods of aluminum foam samples

Denseness of aluminum sample was measured using Archimedes' principle [14]. Porousness was calculated as the percentage value of the difference between the denseness of non-foamed metallic aluminum and the denseness of aluminum foam, relative to the denseness of non-foamed aluminum [15]. For determining the heat conductivity of closed-cell aluminum froth, the guarded hot plate method (ASTM C177) was used. Standard method for measuring the compression resistance of aluminum froth was ASTM C365, involving the use of uniaxial compression testing. The water uptake of the foam was determined using the standard method ASTM C272:2024 and the microstructural peculiarities of samples were identified with Biological Microscope model MT5000.

3. RESULTS AND DISCUSSION

3.1 Results

The main parameters of the microwave-assisted experimental heating/expanding process are shown in Table 1.

Table 1. Main parameters of the microwave-assisted process

Parameter	Version 1	Version 2	Version 3	Version 4
Precursor amounts (g)				
- aluminum powder	97	94	91	88
- $CaCO_3$	3	6	9	12
- total (dry)	100	100	100	100
- water added	10	10	10	10
- total (wet)	110	110	110	110
Aluminum froth amount (g)	97.0	97.1	97.1	97.1
Final heating temperature (°C)	748	750	751	752
Heating time (min)	11.3	11.6	12.0	12.8
Heating rate (°C·min^{-1})	64.4	62.9	60.9	58.8
Specific energy consumption (kWh·kg^{-1})	1.21	1.24	1.29	1.37

Due to the use of microwave irradiation, the heating process of the precursor mixture is very fast, the heating rate varying in the range of 58.8-64.4 °C·min^{-1}. The final temperature of the process is within the limits of 748-752 °C and the duration required to complete it is very short (under 13 min). The energy consumption is at a very efficient level, values reported being in the range of 1.21-1.37 kWh·kg^{-1}.

Appearance of aluminum froth surfaces corresponding to the four specimens is shown in Figure 2. Increasing the content of $CaCO_3$ as an expanding agent from 3 to 12 g of the total dry mix of 100 g clearly influenced this physical appearance of specimens. The homogeneity and uniformity of the closed-cell distribution are kept at an excellent level in all versions, but the cell size exhibits a constant increasing trend.

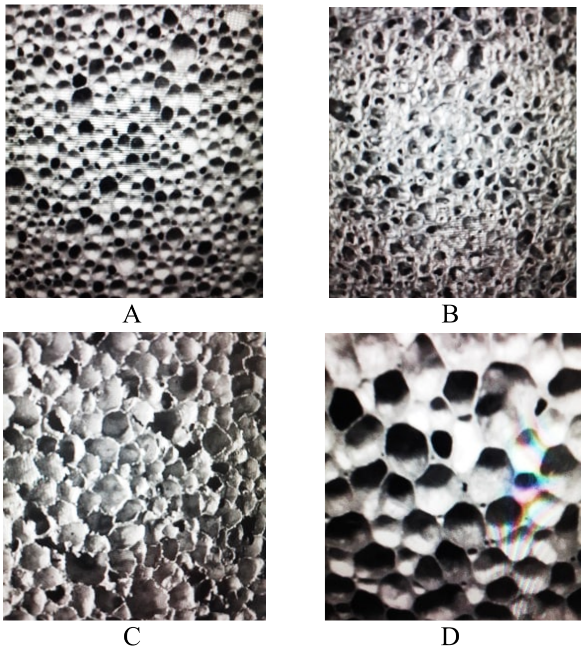
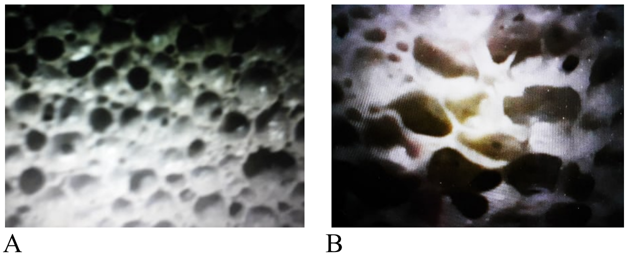


Figure 2. Surface appearance of aluminum foam specimens

A – version 1; B – version 2; C – version 3; D – version 4.

The result of investigating the microstructural features of the four aluminum foam specimens is presented in Figure 3. The images shown in this figure allow the determination of the cell size ranges of the froth specimens. The smallest sizes (between 0.2-0.9 mm) correspond to sample A made by version 1 and the largest (between 1.2-3.0 mm) correspond to sample D made by version 4. The other cell sizes of samples B and C are shown in Table 2.



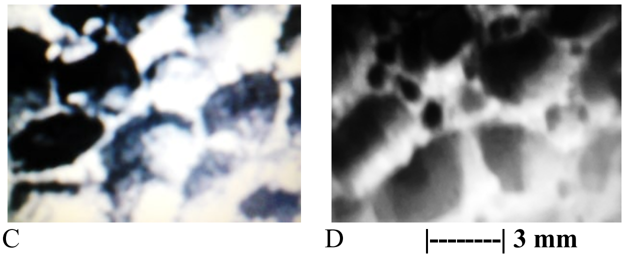


Figure 3. Microstructural appearance of aluminum foam samples

A – version 1; B – version 2; C – version 3; D – version 4.

The investigation results of the physical, mechanical, and thermal features of closed cell-aluminum foam specimens are presented in Table 2.

Table 2. Investigation results of closed-cell aluminum foam specimen features

Version	Apparent denseness ($g\cdot cm^{-3}$)	Porousness (%)	Heat conductivity ($W\cdot m^{-1}\cdot K^{-1}$)	Compression strength (MPa)	Water uptake (vol. %)	Cell size (mm)
1	0.82	66.3	5.58	7.3	0.2	0.2-0.9
2	0.70	73.1	4.80	6.0	0.2	0.5-2.3
3	0.51	78.5	4.03	4.9	0.1	0.9-2.8
4	0.45	80.8	3.23	3.8	0.1	1.2-3.0

Physio-mechanical and heat features of closed-cell aluminum froth samples experimentally made are generally quite similar to other materials of this type presented in the literature. Apparent denseness values of samples fell within normal limits for closed-cell metal foams, i.e. between 0.45-0.82 $g \cdot cm^{-3}$ [16]. Accordingly, the porousness of aluminum samples had values between 66.3-80.8 % and the heat conductivity was in the range of 3.23-5.58 $W \cdot m^{-1} \cdot K^{-1}$, the lowest value corresponding to the specimen obtained using the highest amount of $CaCO_3$. The compression resistance fell within a fairly wide range of values (between 3.8-7.3 MPa), the highest of which corresponding to the highest denseness sample. Water uptake was negligible, as in all aluminum froth specimens.

3.2 Discussion

The main peculiarity of the procedure described in this paper is the adoption of the unconventional technique of direct heating with electromagnetic waves. Although known since the middle of the previous century, the applications of this technique have focused mainly on transmissions and radar, while in the field of heating processes they have been limited to drying and relatively low level-heating. The remarkable energy efficiency of converting electromagnetic wave power into heat upon contact with microwave-susceptible materials is recognized by specialists [17], but its application in industrial thermal processes is still delayed [18].

The specific consumption of the microwave-assisted heating process was between 1.21-1.37 $kWh \cdot kg^{-1}$, but it could be significantly reduced due to the small amount of raw material reported to the experimental oven power. Unlike conventional heating techniques, the microwave-based process is environmentally friendly, with a very low carbon footprint.

The chosen expansion agent ($CaCO_3$) over TiH_2 was adopted due both its more economical price and the increased degree of danger during use and the handling difficulties characteristic of TiH_2. Practically, the adoption of $CaCO_3$ as a foaming agent did not have negative effects on the quality of the aluminum froth, the results being considered satisfactory.

4. CONCLUSION

The current work presents qualitatively improved versions of previous experiments conducted by authors on microwave-assisted closed-cell aluminum foam. Due to the application of the own method of producing fine aluminum powder (below 10 μm) by atomizing the recycled aluminum waste melt with concentrated nitrogen jets, the density of the metal foam was reduced to 0.45-0.82 $g \cdot cm^{-3}$, while porosity increased to 66.3-80.8 %. The compression strength was maintained within quite high limits (3.8-7.3 MPa). The cell size that characterized the four closed-cell aluminum foam specimens had values in different ranges, from 0.2-0.9 mm to 1.2-3.0 mm. Any of these specimen versions are usable depending on the specific surface area requirement. Energy absorption, sound absorption, electromagnetic shielding, and vibration damping are the main abilities of metal froths.

5. REFERENCES

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